(12) UK Patent Application (19) GB (11) 2 140 031 A

(43) Application published 21 Nov 1984

(21) Application No 8313797

(22) Date of filing 18 May 1983

(51) INT CL[3]
C25D 11/26

(52) Domestic classification
C7B 124 684 776 EC
U1S 2060 C7B

(56) Documents cited
GB 1006536 GB 0958669

(58) Field of search
C7B

(71) Applicant
Standard Telephones and Cables Public Limited Company (United Kingdom),
190 Strand, London WC2R IDU

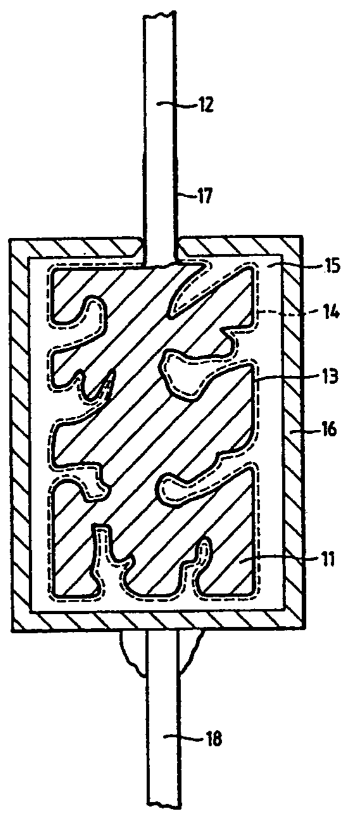
(72) Inventors
Miles Patrick Drake
Ernest James Workman

(74) Agent and/or Address for Service
S R Capsey,
S T C Patent Department, Edinburgh Way, Harlow, Essex CM20 2SH

(54) **Anodic oxidation of tantalum**

(57) An anode body 11 from a tantalum solid electrolytic capacitor is anodised at a temperature below 50°C. It is then treated to a further anodisation at a reduced voltage and for a short period up to 200 seconds at 85° to 100°C. This reduces the leakage current of the anode without introducing the risk of field crystallisation.

Fig. 1.



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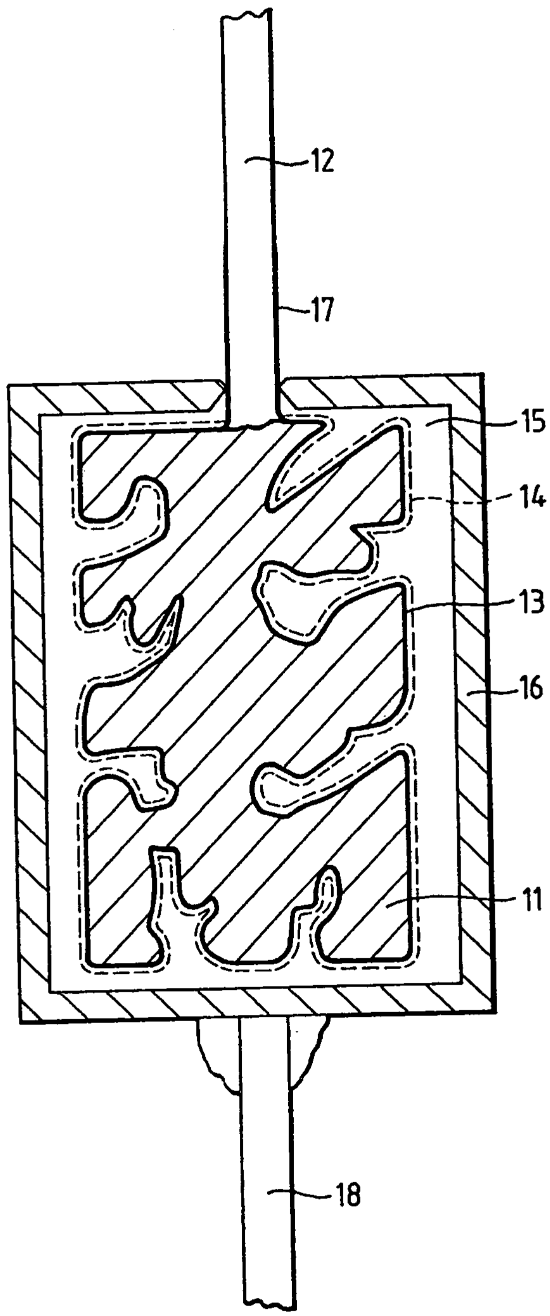
The drawings originally filed were informal and the print here reproduced is taken from a later filed formal copy.

Fig. 1.



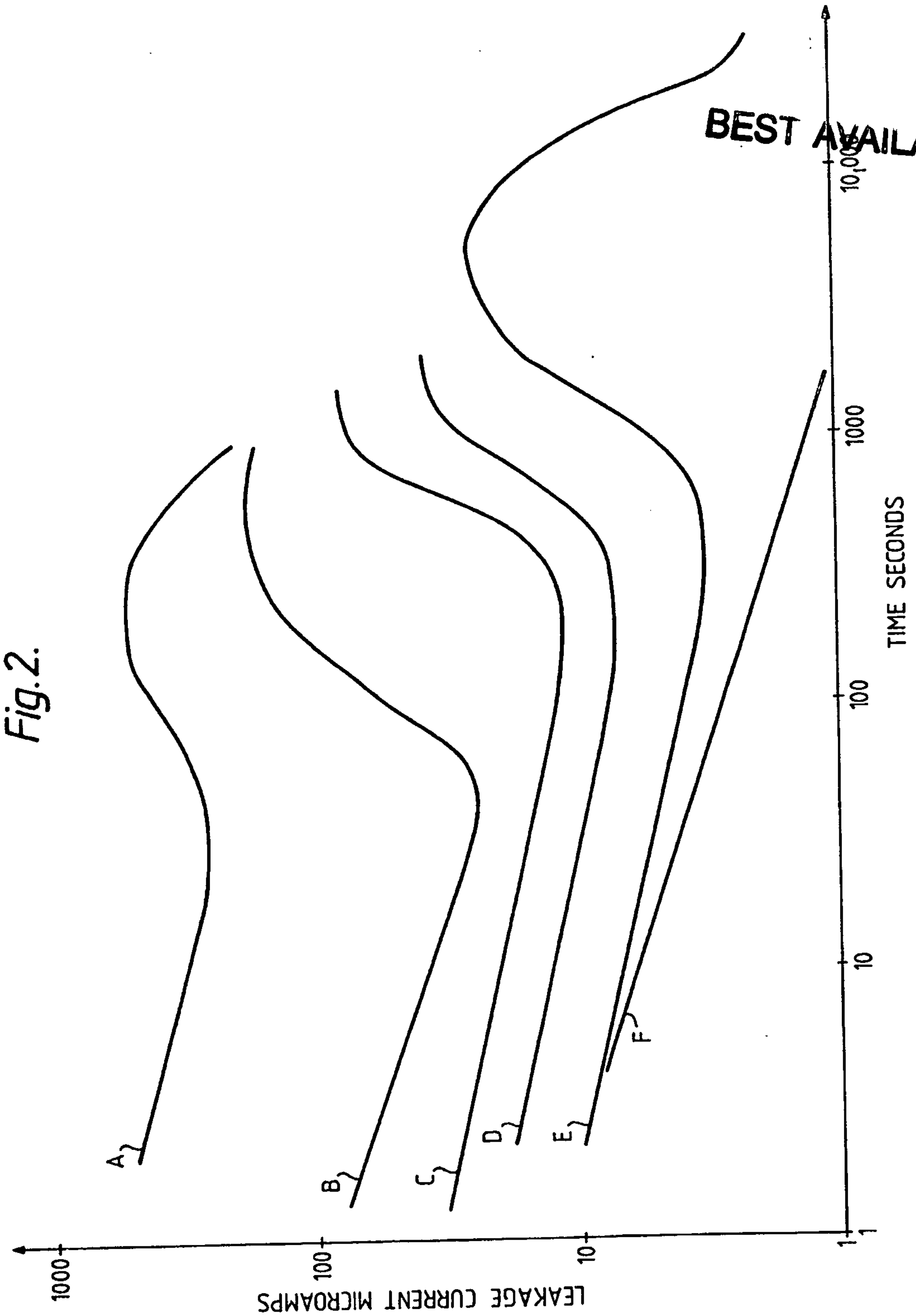

A
B
C
D
E
F
1000
100
10
1
LEAKAGE CURRENT MICROAMPS
1
10
100
1000
10,000
TIME SECONDS

Fig. 2.

SPECIFICATION

An dic xidati n f tantalum

This invention relates to a process for the anodic oxidation of tantalum and in particular to a process for forming capacitor anodes in the manufacture of tantalum by electrolytic capacitors.

The tantalum solid or dry electrolytic capacitor is widely used in a variety of applications where its combination of high capacitance and small physical size are of considerable advantage. These capacitors comprise a porous sintered tantalum anode body whose surface is anodised to form an oxide film. This oxide film provides the capacitor dielectric.

The anodes are pressed from tantalum powder, the processing and the surface condition of which determines the anode surface area and hence the capacitance of the finished capacitor. Recently the so-called 'high gain' tantalum powders have been introduced. These powders, which contain phosphorus, have a very high surface area and thus provide a high capacitance per unit volume.

A disadvantage of these 'high gain' powders is their tendency to exhibit excessively high leakage currents. Attempts have been made to overcome this problem by extending the anodisation process at a high temperature, e.g. above 70°C. It has been found however that the extended period for which the anode is maintained at a high temperature during the anodisation process introduces a serious risk of field crystallisation.

The object of the present invention is to minimise or to overcome these disadvantages.

According to the invention there is provided a process for anodising a tantalum capacitor anode body, the process including anodising the body at a temperature below 50°C, and further anodising the body at a reduced voltage and at a temperature of 85°C to 100°C for a period of up to 200 seconds.

As the anode body is exposed to a high temperature for a short period only the risk of field crystallisation is substantially eliminated. The short high temperature anodisation significantly improves the integrity of the dielectric film without producing an excessive increase in thickness. Thus the electrical leakage of the film is reduced without adversely affecting the capacitance of the finished capacitor.

An embodiment of the invention will now be described with reference to the accompanying drawings in which:–

Figure 1 is a schematic cross-sectional vi w of a tantalum solid electrolytic capacitor;

and *Figure 2* illustrates th leakage current characterisitics of tantalum anod bodi s treated by the process d scribed herein.

Ref rring to Fig. 1, a tantalum solid electrolytic capacitor typically comprises a porous anode body 11 formed by pressing and sintering in vacuum eith r tantalum metal powder r a powdered refractory material coat d with tantalum. Contact t th anode body 11 is provided by a tantalum wire 12 embedded therein. The surface of the body is coated with a dielectric layer 13 of anodic oxide whose thickness determines the capacitance value of the capacitor. This oxide layer 13 is in turn coated with a semiconductive layer 14 typically comprising manganese dioxide formed by thermal decomposition of manganous nitrate. The various techniques for forming this semiconductive layer will be apparant to those skilled in the art.

The anode structure is surrounded by a quantity of graphite 15 contained in a metal, e.g. aluminium, can 16 providing the cathode terminal of the capacitor and contacted by lead 18. Shorting between the capacitor terminals is prevented by an insulating sleeve 17 surrounding the anode lead 12 at the point where this lead protrudes through the can 16.

The dielectric surface layer 13 on the anode body is provided by the following technique.

The pressed and sintered anode bodies are anodised in dilute phosphoric acid at a temperature of 50°C or less. The anodisation is preferably a constant current process so that the voltage rises until a predetermined maximum value is reached at which the current is allowed to fall. Typically the current density for this stage of the process is 35 to 45 mAg^{-1} and the limiting voltage is 70 to 100 volts. After this forming stage the anodes are then subjected to a short further forming by a second anodisation for a period of 30 to 200 seconds in dilute phosphoric acid maintained at a temperature of 85° to 100°C. The effect of this further or secondary forming stage is to enhance the integrity of the dielectric surface layer and thus significantly reduce the leakage current. The results of this treatment are illustrated in Fig. 2 of the accompanying drawings. Under an applied voltage leakage current slowly decreases with time and then rises again after about 100 seconds. As can be seen from Fig. 2 a successive increase in the secondary anodisation period provides a corresponding decrease in the leakage current. Current A shows the leakage time characteristic of an anode treated by the conventional high temperature process. Current B is the characteristic of an anode formed by anodising at 50°C only whilst curves C, D, E and F show the effects of an anodisation at 50°C followed by treatment at 85°C for periods of 10 sec, 20 sec, 30 sec and 40 sec r spectively. As can b seen the two stag treatment provides a significant decrease in the leakag current. In each case the leakage was measured at embient temperature with an applied voltag of 70 volts. We have also found that for peri ds in xcess of 40 seconds no subsequent rise in leakage current is observed. However this period sh uld not b xt nded

beyond 200 seconds as the risk of field crystallisation may th n b r introduced.

CLAIMS

1. A process for anodising a tantalum capacitor anode body, the process including anodising the body at a temperature below 50°C, and further anodising the body at a reduced voltage and at a temperature of 88° to 100°C for a period of up to 200 seconds.

2. A process as claimed in claim 1, wherein said anode comprises a sintered body of a refractory powder coated with metallic tantalum.

3. A process as claimed in claim 1 or 2, wherein said body is anodised in phosphoric acid.

4. A process as claimed in claim 1, 2 or 3, wherein said first anodisation process is a constant current process.

5. A process as claimed in claim 4, wherein the current density is 35 to 45 mAg^{-1}.

6. A process as claimed in claim 4, wherein the anodisation is carried out at a limiting voltage of 70 to 100 volts.

7. A process for anodising a tantalum capacitor anode body substantially as described herein with reference to the accompanying drawings.

8. A tantalum anode body treated by a process as claimed in any one of claims 1 to 7.

9. A tantalum solid electrolytic capacitor incorporating an anode body as claimed in claim 8.

Printed in the United Kingdom for
Her Majesty's Stationery Office, Dd 8818935, 1984, 4235.
Published at The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.